

Chemical characterisation and durability assessment of torrefied radiata pine (*Pinus radiata*) wood chips



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ABSTRACT

Wood chips from New Zealand grown radiata pine were torrefied at 220, 260, and 300 °C with the aim of understanding the fundamentals behind the enhanced durability against major decay fungi. Chemical analysis methods, including high-resolution synchrotron-based X-ray diffraction, were used to ascertain the mode of chemical changes in wood after various torrefaction levels. Compositional analysis showed that the carbohydrates and lignin in 220 °C samples remained at about the same levels as in the control, with a noticeable drop in the relative ratio of carbohydrates levels and an increase in lignin levels at 260 °C, and a steep drop in carbohydrates and a sharp increase in lignin concentrations at 300 °C. Hemicelluloses were the most severely affected carbohydrate component, particularly with 300 °C treatment. Analysis with FTIR showed similar spectra for control and 220 °C treated chips, a broad shoulder around 1610 cm⁻¹ at 260 °C, and several changes at 300 °C involving carbonyl at 1705 cm⁻¹ and c=c and c-c absorptions. Synchrotron-based X-ray diffraction showed a slight contraction of peak *d*(200) for torrefied samples, which is related to increasing hydrophobic cellulose crystallinity. The information obtained suggests that the durability enhancement achieved against *Oligoporus placenta* and *Trametes versicolor* at 260 °C can be attributed mainly to depletion of hemicelluloses, and complete resistance at 300 °C is likely to be related to changes in all cell wall components, particularly hemicelluloses and lignin.

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1. Introduction

Torrefaction is a thermochemical process whereby woody and other lignocellulosic biomass is subjected to temperatures generally ranging between 200 and 320 °C under an oxygen free environment. There is an enormous potential for use of heat-modified wood in generating clean energy (Kiel, 2007) and in developing high-performance products with improved durability and dimensional stability (Esteves and Pereira, 2009; Guller, 2012).

Chemical changes in lignocellulose cell walls are to a large part related to temperature and time of torrefaction. Initial changes at higher temperatures are rapid decomposition of hemicelluloses, which are most unstable with respect to elevated temperatures (Boonstra and Tjeerdsma, 2006), and readily depolymerised at temperatures between 200 and 230 °C (Inari et al., 2007). Changes in cellulose and lignin are comparatively slower and the reactions are dependent upon the temperature and nature of tested materials (Chen and Kuo, 2011). Weight loss of different biomass materials

was highlighted in a separate study by the same investigators (Chen and Kuo, 2010); their work indicated that light torrefaction (240 °C) affected hemicelluloses but essentially not cellulose and lignin, and lignocellulosic material was drastically depleted by severe torrefaction treatment at 275 °C. Yan et al. (2009) found a reduction in mass yield of the biomass with increasing torrefaction temperature, which resulted in greater densification of the biomass with increased carbon content and decreased oxygen content. Chemical characterisation of loblolly pine torrefied at 250 and 300 °C (Ben and Ragauskas, 2012) suggested that the hemicellulose fraction of wood torrefied at 250 °C was completely absent, with cellulose and lignin remaining largely intact, and in the wood torrefied at 300 °C hemicelluloses and cellulose were completely absent, with residues containing modified lignin represented by complex condensed aromatics.

Torrefaction is an attractive approach for improving a range of properties of raw lignocellulosic biomass, depending upon the temperature and time of heat treatment and the type of lignocellulosic biomass. Improvements can include a marked reduction in moisture content (Sadaka and Negi, 2009), and a consequent increase in hydrophobicity, which can lead to durability enhancement. For example, torrefaction in the moderate (250 °C) to high

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(300 °C) temperature range can lead to marked improvements in wood durability (Mohareb et al., 2011). An assessment of heat treatment on wood durability has also been made in several other studies (reviewed in Esteves and Pereira, 2009).

Torrefaction of radiata pine is a major focus of our current research, which is aimed particularly at more efficiently utilising low-quality juvenile wood and wood residues. Heat modification shows promise in enhancing durability of wooden products against common decay fungi (Esteves and Pereira, 2009; Mohareb et al., 2011; Bazzyar, 2012), and it has the potential to replace or minimise use of traditional toxic-based biocides for wood protection. The information presented herein, based on chemical characterisation and durability assessment of radiata pine chips torrefied at 220, 260, and 300 °C, should be helpful for designing high-performance products from heat-treated wood and wood residues, such as composite products. Although previous studies (Dubey, 2010) have provided some information on the durability of heat-treated radiata pine grown in New Zealand, the work reported here combines more complete chemical characterisation, supplementing conventional methods with the high-resolution synchrotron-based X-ray diffraction technique, with durability assessment of the torrefied wood, which can be useful in developing strategies for more effective use of wood chips as a low-value waste material. In wood science research, synchrotron-based X-ray diffraction continues to provide valuable fundamental information, such as on cellulose crystallinity of cell wall layers in developing xylem (Müller et al., 2002) and has played a vital role in unravelling molecular mechanism of cell wall recovery after irreversible deformation (Keckes et al., 2003). Here we extend the application of this powerful technique to characterise high temperature treated wood, where information based on this technical approach is very limited (Zickler et al., 2006).

2. Materials and methods

2.1. Torrefaction

A test rig to conduct torrefaction experiments was constructed. It consisted of an electric furnace heating a steel reactor vessel. The reactor vessel was fed with Argon gas to provide an inert, low

oxygen atmosphere (Fig. 1). The Argon was preheated in a copper coil inside the furnace before entering the reactor vessel. Gas flow was set at 2 L/min. Temperature probes recorded the internal and surface temperature of the reactor vessel, and were used to control the heating from the furnace.

Air dried *Pinus radiata* wood chips obtained from the outer part (sapwood/sawmill slab wood) of freshly felled saw logs (from trees approximately 27 years old milled at Kiwi Lumber, Putaruru, NZ) were used. Torrefaction was carried out at 220, 260 and 300 °C for 30 min with this equipment (Fig. 1). The torrefied material undergoes a number of physical changes, colour change being visibly most noticeable (Fig. 2). Physically the torrefied wood is brittle and dry with reduced bulk density (Rousset et al., 2011). Brittleness and grindability varied with the severity of the torrefaction treatment (Ibrahim et al., 2012).

The wood chips used in this study were of the type typically created at sawmills (25–30 mm wide, 35–40 mm long, and 2–4 mm thick). Chips used in the experiments were selected to be uniform in size. The moisture content of the material going into the torrefaction unit was 12% wet basis. Immediately after torrefaction the moisture content was typically less than 0.5% wet basis. The torrefied material in chip form left exposed to the atmosphere was returned to a moisture content of 6–8%. Ash content was typically 0.5% or less.

2.2. Compositional analysis of torrefied wood

Compositional analysis was conducted using five replicates per torrefaction temperature. Individual samples were ground to 30-mesh (~0.6 mm) and then extracted in dichloromethane using Soxtec apparatus with a boiling time of 30 min and a rinsing time of 1 h. The samples were air-dried and placed in an oven at 55 °C overnight before the lignin and carbohydrate analysis was performed based on the method of Pettersen and Schwandt (1991). For the lignin and carbohydrate analysis, the samples were digested using 72% sulphuric acid in a water bath at 30 °C for 1 h. They were then diluted to 3% sulphuric acid and autoclaved for 1 h at 121 °C and 15 psi, and left to cool before being filtered onto pre-weighed GFC paper for acid-insoluble lignin. The filtrate was kept for acid-soluble lignin and carbohydrates. For acid-soluble lignin, absorbance was measured at 205 nm on a UV/Vis spectrophotometer. The

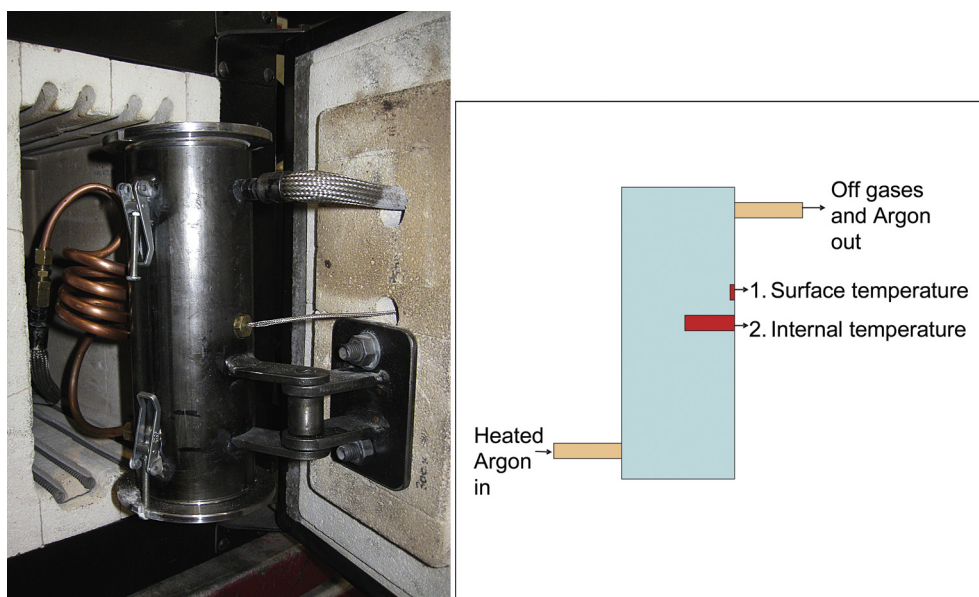


Fig. 1. Reactor tube and gas lines on torrefaction test rig (left), and diagram of temperature probe placement (right).



Fig. 2. Untorrefied *P. radiata* chips (left) and torrefied chips at 300 °C (right).

carbohydrate analysis was performed by diluting the filtrate, adding an internal standard, fucose (Sigma Aldrich), 0.45- μ m filtering, and running on a Dionex IC3000 instrument with eluent generation at 2 mM KOH.

2.2.1. Statistical analysis

A two-way analysis of variance was conducted and least significant difference tests were used to compare differences between various compositions, as well as the differences between compositions at various torrefaction levels. Differences were considered to be significant at $P \leq 0.05$.

2.3. FTIR analysis

Fourier transform infrared spectra of untorrefied wood chips (control) and those torrefied at 220 and 300 °C for 30 min were measured. Sample preparation involved preparing a disk containing a mixture of potassium bromide and the test sample. The mixture was ground before it was placed in a small pressurised die to form a disk. Spectra were recorded at a resolution of 4 cm^{-1} using a Bruker Tensor 27 instrument. A typical spectrum was obtained with 16 scans.

2.4. X-ray diffraction

X-ray diffraction patterns of untorrefied wood chips (control) and those torrefied at 220 and 300 °C were measured using SAXS/WAXS beam-line at the Australian Synchrotron.

Specimens were encased in adhesive tape (Kapton[®], Tesa 51408 from Beiersdorf AG, Hamburg, Germany) immediately prior to mounting for WAXS to minimise moisture loss or gain by diffusion through the tape. A beam energy of 15 keV was used, providing an X-ray wavelength of 0.826561 Å. A Mar-CCD detector (2048 × 2048 pixels) was located 120.714 mm from the specimen, allowing detection of 2θ angles to approximately 35°. The beam size was

0.2 mm by 0.1 mm, and the detector pixel size was 0.079 mm. The exposure time was 2 s, with three points taken per specimen, spaced 1 mm apart to obtain three diffraction patterns per specimen. Values of (centre of the mass of peaks $d(1\bar{1}0)$ and $d(110)$), $d(200)$, and $d(004)$ were calculated from peak maxima. Bragg's equation was used to calculate all values of d . Final conclusions were made at the 95% confidence interval.

2.5. Decay testing

The in-house laboratory "Sutter trial" decay testing method (Sutter, 1978) based on European Standard EN 113 (1989) was used to determine the durability of the four types of wood chips. The following fungi were used with 10 replicates per treatment and fungus:

- *Oligoporus placenta* (New Zealand Forest Research isolate 07/02), a brown-rot fungus that tends to target hemicellulose and cellulose
- *Trametes versicolor* (CTB 863 A), a white-rot fungus that tends to target lignin, cellulose, and hemicelluloses.

The Sutter trial was used due to small-dimension (40 × 30 × 4 mm) wood chip samples. Unlike the EN 113 test, where larger (50 × 20 × 20 mm) samples are used and exposed with test fungi for 16 wk, in the Sutter trial, samples were exposed to test fungi for 6 wk.

Chips from each group were subjected to leaching for 14 days. Leaching involved the re-saturation of samples with water, after which they were placed in water nine times their volume, and the water was changed every 2 days. Subsequent to leaching, the samples were air-dried on racks for 1 wk and then conditioned to a constant weight at 12% equilibrium moisture content (EMC). Chips were then weighed, packaged, sterilised by exposure to ethylene oxide gas, and then transferred aseptically into prepared sterile

Table 1
General composition of wood and torrefied wood.

Samples	Samples (g) per 100 g oven dried samples			
	Carbohydrates	Lignin	Extractives	Un-hydrolysed materials
Wood chips (control)	65.85 ± 0.01 ^a	27.40 ± 0.32	1.00 ± 0.12	6.01 ± 0.20
Torrefied chips 220 °C 30 min	62.80 ± 3.25	28.32 ± 0.49	1.21 ± 0.19	7.66 ± 3.93
Torrefied chips 260 °C 30 min	59.83 ± 2.01	34.85 ± 0.21	0.99 ± 0.11	4.31 ± 1.91
Torrefied chips 300 °C 30 min	28.15 ± 1.84	67.71 ± 0.79	1.44 ± 0.10	2.69 ± 1.15

^a ± value represents standard deviation of mean ($n = 5$).

containers (Sutter jars). The Sutter jars contained 2% malt agar and had an active growth of the test fungi inoculated 2 wk prior to sample placement. The samples were incubated for 6 wk at 25 °C. After incubation, the chips were brushed carefully to remove any adhering mycelium and placed in containers to air-dry, reconditioned to constant weight at 12% EMC, and reweighed. Percentage weight loss for each sample was calculated and means were determined for each fungus/sample combination.

3. Results and discussion

3.1. Compositional analysis

Compositional changes in wood components after torrefaction are summarised in Table 1, with a detailed analysis presented in Table 2. The ratio composition of the wood changed significantly ($P \leq 0.05$) with increasing temperature (severity) of the torrefaction. The carbohydrates and lignin in wood torrefied at 220 °C remained at the same level as in untreated wood chips. While they changed significantly as the treatment temperatures increased, there was a noticeable drop in the relative amount of carbohydrates and an increase in amount of lignin at 260 °C, and a steep drop in the carbohydrates and increase in lignin at 300 °C.

The extractive level for the 300 °C samples was similar to that for the control sample. However, compared to the control, there was an increase and a drop in the extractive levels for 220 °C and 260 °C samples, respectively, which is difficult to explain. A consistent trend was also not found for acid-soluble lignin. However, predictable results were obtained for acid-insoluble lignin, with the level of lignin for the 220 °C sample being similar to that for the control sample, which reflects corresponding decreases in the level of all hemicelluloses. Similar levels of glucan were found for control, 220 °C, and 260 °C samples, but there was a decrease in the level for the 300 °C sample, which suggests modifications also in cellulose at torrefaction temperatures exceeding 260 °C (Chen and Kuo, 2011). Whereas the levels for the hemicelluloses arabinan, galactan, xylan, and mannan for the control and the 220 °C sample were similar, the level decreased slightly at 260 °C and significantly greatly at 300 °C ($P \leq 0.05$).

Table 2
Specific composition of extractives, lignin, and various carbohydrates before and after torrefaction.

Sample	%w/w oven dried sample	% w/w extracted oven dried sample						
	DCM extractives	Lignin		Neutral carbohydrates				
		Acid-insoluble (Klason)	Acid soluble	Arabinan	Galactan	Glucan	Xylan	Mannan
Wood chips (control)	0.53	26.73	0.66	1.27	2.08	45.50	5.47	11.30
Torrefied chips 220 °C	0.75	27.50	0.75	1.07	2.32	43.40	4.90	11.00
Torrefied chips 260 °C	0.30	34.00	0.95	0.45	1.41	47.10	3.25	7.72
Torrefied chips 300 °C	0.55	67.05	0.66	0.02	0.09	27.10	0.22	0.70

It is well known that during torrefaction some of the solid mass of the wood is lost as gases (mainly CO₂ and CO) and liquids (acetic acid, water), with losses initially occurring in the hemicellulosic components (Prins et al., 2006; Pommer et al., 2010). This explains the change in the proportions of carbohydrates and lignin, particularly at temperatures exceeding 260 °C. The effect of temperature on wood components varies, with hemicelluloses starting to break down around 225 °C and changes in lignin occurring at temperatures exceeding 250 °C (Kiel, 2007).

3.2. FTIR analysis

While compositional analysis provided information on changes in the level of major cell wall components in relation to temperature treatment of the substrate, FTIR analysis was useful in characterising specific chemical changes occurring in these components (Fig. 3).

There were very little differences in the IR spectra of untorrefied wood chips and chips torrefied at 220 °C (TC220 °C). A broad shoulder was discernible around 1610 cm⁻¹ in the TC220 °C sample. However, there were many changes between chips and TC300 °C. There was a broad carbonyl at 1705 cm⁻¹, possibly representing a resonance canonical (carbonyl) form of furan, strong broad C=C absorptions (1608, 1516, 1642 cm⁻¹), which were the dominant differences in the spectra, and a weak C–C band at 1213 cm⁻¹.

Xyloses (5 C sugars) will degrade to compounds like furfural (<250 °C), which in turn is converted into furan-type structures (250–300 °C) (Table 3).

Hemicellulose is a complex, branched, and heterogeneous polymeric network, based on pentoses (5 C sugars) such as xylose and arabinose, hexoses (6 C sugars) such as glucose, mannose, and galactose, and sugar acids. During thermolysis, hemicellulose is partly depolymerized by hydrolysis and/or thermal chain scission to provide “reacting hemicellulose” (Bourgeois and Guyonnet, 1988). This intermediate is decomposed by acid and radical reactions to yield many substances (e.g., furfural) that recombine to form torrefied hemicellulose. Water and acids are formed during these reactions and are released into the reaction environment. Some of this water may be reused to depolymerize hemicellulose or to release acids from the hemicellulose by hydrolysis of acetate groups. Acids can also be formed by radical reactions. The water and acids released from hemicellulose become available to further depolymerize cellulose and lignin (Boonstra and Tjeerdma, 2006; Dubey, 2010). The results from FTIR analysis in our work add specific chemical information on torrefied wood from radiata pine grown in New Zealand (Table 3), providing further support for proposed interpretations of chemical changes occurring during torrefaction of lignocellulosic substrates.

3.3. X-ray analysis

Synchrotron-based X-ray diffraction provides high-resolution diffractograms that overcome many shortcomings of conventional X-ray sources used in the study of wood (Leppanen et al., 2009). A

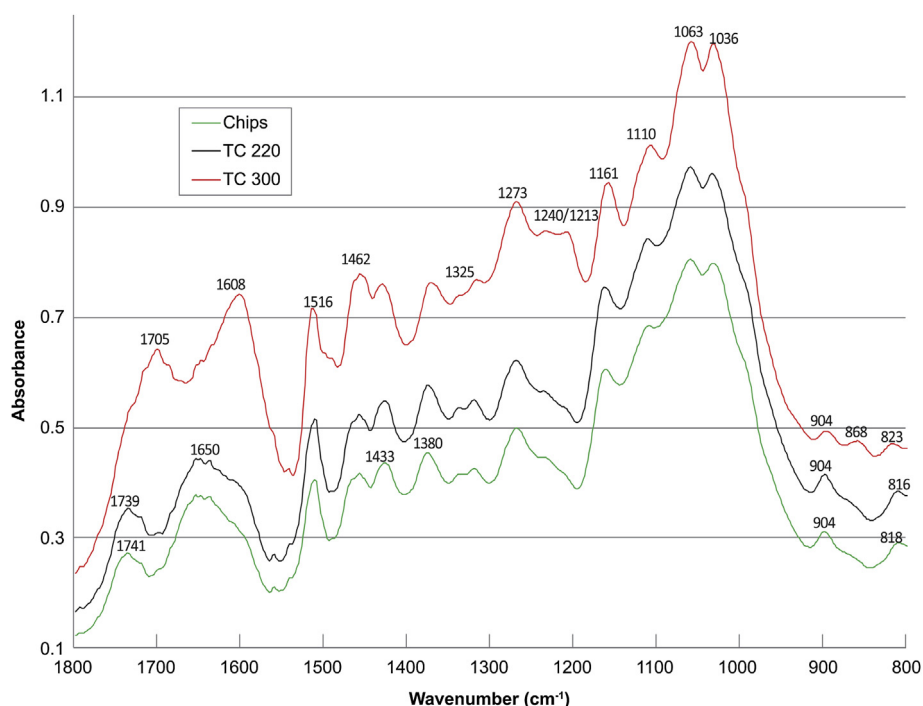


Fig. 3. FTIR spectra of untreated wood (chips), and torrefied chips at 220 °C (TC220) and 300 °C (TC300).

WAXS diffractogram of wood chips used as a control, and wood torrefied at 220 °C and 300 °C for 30 min, showed strong peaks assigned to (1 $\bar{1}$ 0), (110), (200), and (004) planes of cellulose (Fig. 4).

A slight contraction in the centre of mass of peaks $d(1\bar{1}0)$ and $d(110)$ at 220 °C and 300 °C as compared to the control has been noted, indicating a change in hydrogen-bonding in the crystal (Hill et al., 2010). As the temperature increases, the peak $d(200)$ contracts by 0.1 Å for the specimen heated at 220 °C and 300 °C, which shows a slight decrease in distance between chains in hydrogen-bonded sheets, which could be related to the increase of hydrophobic cellulose crystallinity (Pétrossans et al., 2003). A small hump at ($2\theta = 14$ ca.) shows a water peak, which is consistent in all samples (Hill et al., 2010). However, no change in the peak $d(004)$ has been noted at 220 °C and 300 °C, suggesting that there is no change in fibre repeat distance. These results are in agreement with the FTIR analyses, and can be explained by assuming that the high torrefaction temperature results in degrading the matrix, and affecting cellulose crystallites as compared to a simple wood-drying process. Nevertheless, the change in distance between chains in hydrogen-bonded sheets could possibly be due to a conformational change in cellulose surface crystals from an inter-molecular hydrogen-bonding to an intra-molecular hydrogen bonding conformation (Hill et al., 2010).

3.4. Decay testing

Mean percentage weight losses after exposure of the chips to test fungi are shown in Table 4. Torrefaction at lower temperatures (220 °C) had limited impact on biological reactivity, as weight losses with *O. placenta* were similar to those for untreated (control)

chips and somewhat lower with *T. versicolor* compared to the control. Much lower weight losses occurred at 260 °C with both *O. placenta* and *T. versicolor*; however, these losses were not sufficiently low for the torrefied wood to be considered inert. Weight losses generally need to be less than 2% for the material to be considered inert in this type of test (Sutter, 1978). Therefore, only

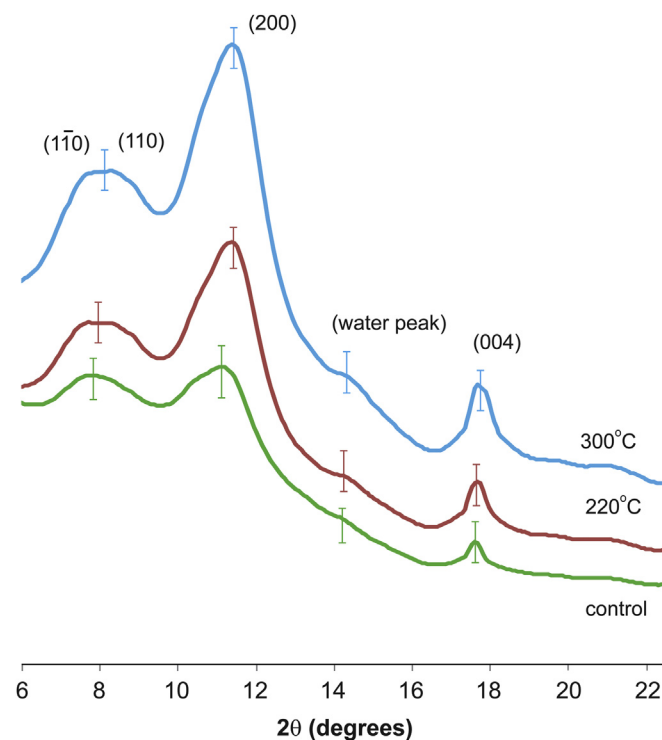


Fig. 4. WAXS diffractograms of wood chips as a control, and wood torrefied at 220 °C and 300 °C.

Table 3
Representative peaks in FTIR.

TC300	Carbonyl	C=C	C=C	C=C	C=C
Furan based peaks	1705	1608	1516	1462	1213

Table 4

Mean percentage weight losses (range in parenthesis) of wood chips exposed to *Oligoporus placenta* and *Trametes versicolor*.

Samples	<i>Oligoporus placenta</i>	<i>Trametes versicolor</i>
Wood chips (control)	33.71 (16.98–44.26)	9.81 (5.46–17.41)
Torrefied chips 220 °C 30 min	34.47 (18.34–52.46)	5.47 (0.19–12.72)
Torrefied chips 260 °C 30 min	10.15 (0.74–19.34)	1.02 (0–3.21)
Torrefied chips 300 °C 30 min	0	0

the material torrefied at 300 °C was found to be biologically inert, judging by zero weight loss with the test fungi.

The results from the fungal decay tests presented showed durability enhancement against both brown-rot and white-rot fungi, and a close correlation was observed between durability increases (as judged by weight loss), treatment temperature, and duration and mass losses, with the higher temperatures of 260 °C and 300 °C resulting in the greatest durability improvements. These results are consistent with the observations made in several other studies. For example, [Welzbacher et al. \(2007\)](#) found that decay resistance for a given treatment intensity was superior at high temperatures.

The information from combined chemical analysis and durability assessment undertaken in our work suggests that durability enhancement in the chips treated at 260 °C is likely to be due mainly to depletion of hemicellulose, and complete resistance of the chips treated at 300 °C to test fungi can be attributed to changes in all cell wall components, especially hemicellulose and lignin. The resistance to fungal decay can be linked to chemical changes in cell wall polymers that occur during torrefaction ([Hakkou et al., 2006](#); [Welzbacher et al., 2007](#); [Šušteršič et al., 2010](#)), the most significant of these being molecular changes leading to depletion initially of hemicelluloses, which are very susceptible to thermal degradation and are readily depolymerised in the temperature range between 200 and 230 °C ([Inari et al., 2007](#)). Cellulose is affected in more advanced stages, with changes also occurring in the structure and composition of lignin, resulting from thermo-cross linking reactions of degradation products. [Weiland and Guyonnet \(2003\)](#) have proposed two reasons for improvements in decay resistance of wood due to heat treatment. One is reticulation of some molecules produced during heat treatment, such as furfural. The second reason, lignin renders wood substrate which is unrecognisable by fungal enzymes. Our FTIR analysis also provided evidence of furfuryl generation at higher temperature ranges. Esterification of cellulose due to acetic acid released from degradation of hemicelluloses may be a factor.

It has been proposed that the composition of torrefied wood, such as the carbon content and O/C ratio, can prove to be a useful signature for predicting decay resistance of a product ([Šušteršič et al., 2010](#)) and can potentially form the basis for engineering products for specific applications ([Tjeerdsma et al., 1998](#)), depending on the temperature and time of treatment. The information obtained in our study, combining bulk chemical analysis with FTIR spectral determination, also points to rapid degradation of hemicelluloses at moderate temperatures, with chemical changes in lignin occurring under more severe temperature conditions.

Of the components of lignocellulosic cell walls, hemicelluloses in their native state are generally considered the most susceptible to degradation by brown-rot fungi ([Irbe et al., 2006](#)), and this may be the reason for substantial weight losses in wood chips that had been torrefied at 220 °C for 30 min prior to exposure to the brown-rot fungus *O. placenta* in our work. The chemical analyses of the substrate in our study showed only minor alterations in hemicelluloses and no changes in cellulose and lignin at 220 °C. Chemical modification and/or depletion in hemicelluloses can adversely affect the rate and extent of degradation of lignocellulosic

cell walls by wood-decay fungi, and particularly brown-rot fungi, which can more readily and extensively degrade hemicelluloses and cellulose than lignin. Extensive losses in hemicelluloses, with some losses in cellulose at 260 °C, and more extensive changes in cell wall components at 300 °C, are considered to be the reason for only a slight weight loss at 260 °C and no weight loss at 300 °C from exposure to *O. placenta* in our study. Brown-rot fungi can preferentially degrade wood polysaccharides but can only slightly depolymerise lignin ([Jin et al., 1990](#); [Jensen et al., 2001](#)). Brown-rot fungi degrade holocellulose, causing rapid depolymerisation of cellulose in early stages ([Koenigs, 1974](#); [Green and Highly, 1997](#)), which results in measurable losses in wood strength before weight losses are detected. Prior to enzymatic attack these fungi deploy small, diffusible extracellular agents ([Koenigs, 1974](#); [Goodell et al., 1997](#)) that can penetrate into lignocellulosic cell walls, rendering them accessible to further degradation by enzymes ([Wang and Gao, 2003](#)). Extracellular H₂O₂ has been cytochemically detected in early stages of brown-rot decay ([Kim et al., 2002](#)), and a role of H₂O₂ in early degradation of cellulose has been suggested. These early-stage decay mechanisms make brown-rot fungi an attractive agent for biotechnological applications, such as for pretreatment in biofuel production ([Ray et al., 2010](#); [Vaidya and Singh, 2012](#)).

It is generally thought that, like brown-rot fungi, white-rot fungi, such as *Phanerochaete chrysosporium* and *T. versicolor*, also deploy highly reactive hydroxyl radicals for fragmentation of wood polymers in advance of enzymatic action, the process being mediated by H₂O₂ ([Kremer and Wood, 1992](#); [Daniel et al., 1994](#); [Tanaka et al., 1999](#)). However, white-rot fungi markedly differ from brown-rot fungi in other aspects of wood cell wall degradation, and *T. versicolor* is an efficient degrader of lignin, causing simultaneous degradation of all cell wall components. The presence of lignin peroxidase and xylanase has been demonstrated during *T. versicolor* degradation of wood cell walls by immunocytochemical labelling ([Blanchette et al., 1989](#)). As lignin is not likely to be modified within the short period (30 min) of heat treatment at 220 °C, the reason for lower wood weight loss (6.49%) than in the control (10.85%) is not immediately clear. It could be that changes in hemicelluloses at the beginning of the temperature treatment of 220 °C have an effect on the rate of cell wall degradation by *T. versicolor*. However, the negligible weight loss at 260 °C and absence of weight loss at 300 °C are probably related to extensive depletion in hemicellulose and thermally induced changes in lignin at higher temperatures ([Rousset et al., 2011](#)). In our work, zero weight loss in the substrate treated at 300 °C for 30 min to both *O. placenta* and *T. versicolor* exposure would point to rapid and extensive modifications in cell wall components, including lignin, at this temperature, as also reported previously ([Ben and Ragauskas, 2012](#)), leading perhaps to lack of accessible substrate.

4. Conclusion

In this study, durability and chemical characteristics of radiata pine wood chips torrefied at 220, 260, and 300 °C were evaluated. Results showed that resistance to fungal degrade after torrefication, with a close relation between durability enhancement and treatment temperature. Chemical analysis revealed that durability enhancement in the wood treated at 260 °C is likely to be due to depletion in hemicellulose. The significant resistance to fungal decay at 300 °C is linked to chemical changes in hemicellulose and lignin, resulting in cell wall polymerization and certain chemicals such as furan being produced during torrefication at this temperature. Synchrotron-based X-ray diffraction showed that the torrefied wood had an increase of hydrophobic cellulose crystallinity, causing decreased wettability, which is directly related to durability enhancement.

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